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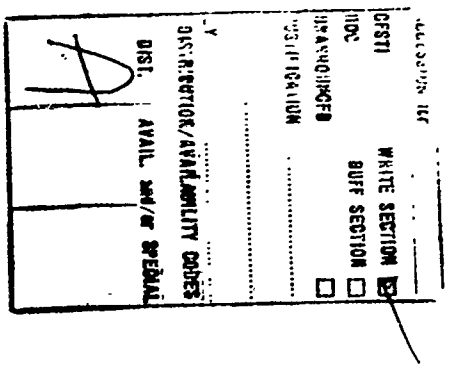

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13 ABSTRACT

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14 KEY WORDS	LINK A		LINK B		LINK C	
	ROLE	WT	ROLE	WT	ROLE	WT
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ELECTRICAL RESISTIVITY						

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Abstract

Low temperature (4 °K - 70 °K) thermal conductivity measurements have been carried out on two copper-aluminum (copper plus 1 at. % and 15 at. % aluminum) which have undergone various stages of fatiguing. From the temperature variation of the lattice component of the measured thermal conductivity, the dominant lattice imperfections introduced by fatiguing were determined. It is found that in the copper plus 1 at. % aluminum alloy, the damage is in the form of dislocation pile-ups. In the copper plus 15 at. % aluminum alloy, the damage is a random distribution of dislocations. This damage is of the same nature as that induced by uniaxial tensile deformation.

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I. INTRODUCTION

In the study of the fatigue of metals under cyclic stress, many methods have been employed to determine the nature of lattice defects introduced. The present paper reports on a study of lattice defects, using the lattice component of the thermal conductivity as an indicator of the dominant lattice defects. The defects commonly considered are dislocations, vacancies and stacking faults. Each of these defects scatters lattice waves according to its own characteristic frequency dependence. This frequency dependence will in turn manifest itself in the temperature dependence of the lattice thermal resistivity of the metal. Hence thermal conductivity measurements at low temperatures have proved to be a useful tool in detecting imperfections in the crystal structure. This method has the merits that sample preparation is relatively easy and that it is non-destructive to the sample so that cumulative damage can be studied. One of its principal limitations is that it can be used only for metals of sufficiently high electrical resistivity, lest the electronic thermal conductivity should overshadow the lattice component.

In this paper, thermal conductivity measurements are reported on two copper-aluminum alloys at several stages of fatiguing. These measurements are carried out in the temperature range 4° - $70\ ^{\circ}K$.

II. SAMPLES

The samples used in these experiments have two different concentrations of aluminum in copper: 1 at.% and 15 at.% . They were supplied by the American Anaconda Brass Company and came as rods of 1/2 inch diameter, approximately 8 inches long. In order to facilitate thermal conductivity and electrical resistivity measurements, the center section of the sample (about 5 inches long,) was machined to a diameter of approximately 1/4 inch. However the ends of the sample had to be kept at the original diameter for mounting purposes on the fatiguing machine. Between each end and the center section there was a short length along which the sample was gently tapered (an hourglass shape). The tapering was done in such a manner that the side view of the sample had curves with radii of curvature of about 1.5 inches. The purpose of this tapering was to reduce the probability of stress concentration at any localized region as stresses were applied. After machining, the samples were polished with several gradations of emery paper. The final polish was done with a #600 paper, with the polishing direction parallel to the length of the sample. Thermal conductivity and electrical resistivity measurements were done on the samples in the following states: annealed for 48 hours at 1000°C, fatigued for 500 cycles, fatigued for 10 kilocycles; the 15 at.% sample was also fatigued for 100 kilocycles. For comparison with the effect of other modes of cold working, a different set of samples were also measured after they had been given a 5% plastic deformation under uniaxial stress. All the fatiguing and deforming were done by the Metallurgy Department at the University of Connecticut.

III. EXPERIMENTAL

The cryostat used for these experiments was a commercial (Cryogenics Associates) variable temperature cryostat. In this cryostat, cooling is provided by the flow of cold helium vapor in a capillary wound around a controlled temperature block. Temperature control is achieved by balancing this cooling by a heater power. Current to the heater is triggered by the unbalanced signal from an AC resistance bridge which senses the resistance of either the germanium (4° - $25\ ^{\circ}K$) or the platinum (25° - 70°) resistors imbedded in the block. Samples were mounted onto the block by means of screws.

For thermal conductivity measurements, a temperature gradient was established along the sample by means of a heater attached to one end of the sample. Temperatures were measured with germanium resistance thermometers. The primary standard of temperature was a pair of resistors that had been calibrated by the manufacturer, Cryo-Cal. Subsequent calibrations of other germanium resistance thermometers were based on these standards. The accuracy of the calibration is approximately 5 millidegrees at low temperatures and 40 millidegrees at high temperatures. Thermometer resistances were determined by measuring the DC potential drop across the resistors with a Hewlett-Packard Model 2401C Digital Voltmeter and a Dymec Model 2411A Data Amplifier; measurements were made for both directions of current flow to reduce the effects of thermal e.m.f.'s . The current through the resistors was determined by similarly measuring the potential drop across a standard resistor. The extremely high input impedence ($> 10^{10}$ ohms) of the data amplifier produced negligible loading of the thermometer resistors.

Thermometer resistance was converted to a temperature by means of an eight-term polynomial expansion of the resistance of the form:
$1/T\ (R_j) = \sum_{n=1}^{6} A_{j,n}(\ln R_j)^n$; the coefficients were determined from the earlier thermometer calibration. Heater voltages and currents were measured with the same electronics; the voltage to be measured was selected by a Leeds and Northrup rotary switch designed for low level DC signal applications.

Electrical resistivities of the samples were measured at the following temperatures: 1.1°, 4.2°, 77° and $273\ ^{\circ}K$. A DC potentiometric method was used, employing a Honeywell Model 2783 Potentiometer and a Keithley 14 Nanovolt Null Detector.

For reasons that will be explained in the next section, thermal conductivity measurements on one of the samples (C3) were also carried out from $1^{\circ} - 4\ ^{\circ}K$. These measurements were done with a different cryostat and a different set of thermometers which were calibrated against He^4 vapor pressure. The cryostat is similar in construction and operation to that used by others in this laboratory (see, for example, Mitchell).[1]

IV. RESULTS

Table I gives the cold work history of the samples, together with the electrical resistivities at various temperatures. The values of the electrical resistivities of the annealed samples agree well with the room temperature values given in the literature.[2] No independent analysis of the composition of the samples was carried out.

The measured thermal conductivity, K , is made up of two parts: the electronic component K_e and the lattice component K_g.

$$K = K_e + K_g \qquad (1)$$

The electronic components is assumed to satisfy the Wiedemann-Franz law, with a small correction (< 1%) due to the ideal thermal resistivity, W_i, of the parent metal, i.e.

$$1/K_e = W_i + \rho_o/L_o T$$

where ρ_o is the residual resistivity, taken to be the value at helium temperatures, L_o is the Lorenz ratio, $2.445 \cdot 10^{-8}$ Ω watt^{-1} deg^{-2}, and W_i is taken from previous measurements of thermal conductivity of pure copper by other workers (e.g., White).[3]

Having determined the electronic part of the thermal conductivity in this way, K_g can be calculated from Eq. (1). Figure 1 shows the variation of K_g with temperature for the 1 at.% aluminum samples. Results for the 15 at.% aluminum samples are shown in Fig. 2. Aside from the error due to the uncertainty in the geometry of the samples, the errors in the values of K_g are estimated to be 3% at the low temperature end and 10% at the high temperature end of the curves in Fig. 1. In Fig. 2, they are 2% and 10% respectively. (A detail error analysis is given in Ref. 4.)

The thermal conductivity by lattice waves is limited by several resistive mechanisms, each contributing in an additive manner to the lattice thermal conductivity W_g. Thus

$$1/K_g = W_g = W_e + W_p + W_s + W_d + W_u \qquad (2)$$

where W_e is the lattice thermal resistivity due to phonon-electron interactions, W_p is due to phonon scattering by point defects, W_s is due to scattering by stacking faults, W_d is due to dislocation, and W_u is the resistivity due to anharmonic three-phonon Umklapp interactions. The

temperature dependences of the various resistive processes are as follows: $W_e \propto T^{-2}$, $W_d \propto T^{-2}$, $W_s \propto T^{-1}$, $W_p \propto T$ and $W_u \propto T$. Thus W_p and W_u will be most important at higher temperatures and are expected to negligible at the lowest temperatures.

To determine the nature of the damage done to the crystals, the lattice conductivity, K_g, of the cold worked samples and that of the annealed samples are compared. It is seen in Fig. 1 that K_g of the annealed sample, C1 varies as T^2 below 15 ^oK. This behavior is consistent with the scattering of phonons by electrons. The value for this samples is 1.1×10^3 cm-deg watt^{-1}, which is in reasonably good agreement with the work of Charsley and Salter[5] on the thermal resistivity of annealed copper-aluminum alloys. For sample C2, C3 and C5, which were cold-worked, the same T^2 dependence can be established between 6^o and 15 ^oK. The additional resistivity can therefore be attributed to dislocations (Klemens),[6] or to phonon scattering by dislocations and by impurity atmospheres around dislocations (e.g. Ackerman and Klemens).[7] The validity of the latter model has been verified for copper-aluminum by Mitchell et. al.,[8] who found that the dependence of thermal resistivity is of the form

$$\frac{W_d T^2}{N_d} = 0.9 + 4.9\, c_o \quad (10^{-7} \text{ cm}^3 \text{ deg}^3 \text{ watt}^{-1}) \qquad (3)$$

where W_d is thermal resistivity due to dislocation, N_d is the dislocation density and c_o is the atomic fraction of aluminum in copper. The numerical coefficients in (3) are obtained from the empirical data of Charsley et. al.[9] The dislocation densities in these samples can then be calculated by (3) from the differences, W_d, in thermal resistivity between these samples and sample

C1. They are given in Table I.

Below 6 ^{o}K, all the cold work samples show a departure from the T^2 dependence, with K_g decreasing more rapidly than T^2. It was suspected at first that this departure may be a result of experimental errors. Thermal conductivity measurements were therefore performed on sample C3 from 1^{o} - 4 ^{o}K with a different cryostat. The result is also shown in Fig. 1. Because K_g becomes a decreasing fraction of the total conductivity as temperature is lowered, uncertainty in the data becomes larger. For this reason, points below 2 ^{o}K are omitted. It is found that the departure from the T^2 dependence is maintained at this lower temperature range also, an indication that the observed decrease in K_g below 6 ^{o}K is a real effect. The temperature dependences of their thermal resistivities are found to be $T^{-2.4}$ for the fatigued samples and $T^{-2.8}$ for samples C5.

These temperature dependences can be ascribed to two components of resistivity: one that varies as T^{-2} and one that varies as T^{-3}. The T^{-2} dependence would be due to dislocations, as discussed earlier. A T^{-3} dependence, according to theory, is characteristic of scattering of phonons by boundaries such as external boundaries, grain boundaries, and sub-boundaries (dislocation walls). However, since sample C1 exhibits a T^{-2} dependence only, it is unlikely that external or grain boundary scattering is important for these samples. Therefore we suggest this increase in thermal resistivity to be due to scattering of phonons from dislocation walls.

Dislocation walls are made up of a number of parallel dislocations grouped together. The strain field of such a group of dislocations will have the effects of enhancing each other so that the combined strain field is stronger

than the sum of the strain fields of individual dislocations at distances greater than the spacing between them. Thus a stronger scattering of long-wavelength phonons results. Note that the average separation between dislocations, randomly arranged and of the densities observed in the present samples, would be in the vicinity of 300 Å. This is comparable to the wavelength of the dominant phonons at 3 °K. This wavelength is a measure of that portion of the strain field around the dislocations which contributes most to the phonon scattering. Consequently one would expect shorter phonons (dominant at higher temperatures) to be scattered as if dislocations act independently, while longer phonons would be sensitive to the inter-dislocation arrangement which modifies the long-range strain fields. One may thus expect some departure from the T^2 dependence below, say 3 °K, if dislocations are almost random, but adjust their mutual positions to minimize the long-range strain energy (e.g. Ackerman and Klemens).[7] On the other hand, if a substantial fractions of the dislocations are grouped in systematic arrays such as walls, with a consequent reduction in dislocation spacing within such a group, the transition region from the T^2 behavior, indicative of single or random dislocations, will be moved to high temperatures (shorter wavelengths).

The question that remains, then, is to determine the structure of the dislocation walls. Here, two possibilities may be considered:
(1) Dislocations are aligned on top of each other, forming a polygonized wall. This happens as a results of interactions between dislocations slipping on adjacent planes (slip polygonization, Friedel).[10] (2) They are piled up against each other along their slip plane. This occurs when dislocations are stopped by an obstacle on the slip plane, such as the grain boundary

(Cottrell).[11] However, structure of the first type are expected to occur in single crystals in the region of easy glide (stage I of working hardening) only. For polycrystals, as our samples are, easy glide is unlikely to take place as dislocations slip is confined within grains so that the slip lines are short. As dislocations are stopped at the grain boundaries, they will pile up against each other and walls are formed. The fact that the temperature variation of K_g for sample C5 is more rapid than that for the fatigued samples seems to indicate that the pile-up is more intense for tensile stress, i.e. there are more dislocations involved in the pile-up.

Above 15 ^{o}K, K_g increases less rapidly with temperature, until at approximately 35 ^{o}K, it decreases again. At 60 ^{o}K, K_g for all the samples appear to merge together. It therefore appears that point defects (mainly vacancies) were not generated in appreciable amounts during cold working of these samples. However, any point defects generated would appear against a background of 1 at.% aluminum and therefore are not likely to be observed. This view is also supported by the electrical resistivity measurements. According to Jongenberger,[12] the increase in resistivity due to vacancies in copper is 1.25 $\mu\Omega$cm/at.% . As shown in Table I, the resistivities of these samples do not vary by more than 0.5% . It is concluded that vacancies are not generated in any appreciable amount, if they are generated at all. On the other hand, stacking faults should give a T dependence to the thermal conductivity and no such dependence is observed for all the samples. This conclusion is also supported by the electrical resistivity measurements. Howie,[13] following the suggestion of Broom that a stacking fault can be considered as made up of two partials that have dissociated from a dislocation, calculated the increase in resitivity due to stacking faults

and found that $\rho_s = 1.10^{-6}\beta$ $\mu\Omega$cm . Here β is the fault density and is estimated to be equal to $2.5\ 10^{-7}\ N_d\ cm^{-1}$, where N_d is the dislocation density. For our samples, β should be of the order of 10^4 , the corresponding increase in resistivity should then be 10^{-2} $\mu\Omega$cm roughly. As shown in Table I, no such increase is observed. Furthermore, stacking fault thermal resistivity can be shown to given by $W_s T = 3.5\ 10^{-4}\ \alpha$ (cm deg^2 watt^{-1}) (Kemp et. al.),[14] where α is the reciprocal of the number of atomic planes per stacking fault. For the value of β estimated above, α should be on the order of 10^{-4}, so that $W_s T = 3.5$ cm deg^2 watt^{-1} . At 50 ^{o}K, W_s is approximately 0.1 cm deg watt^{-1} . This value is two order of magnitude smaller than the measured thermal resistivity and should not be observable at all. Indeed, when dislocations are piled up the mutual interaction between dislocations should prevent the occurrence of dissociation, and the probability that they be observed will be even smaller.

Results of copper plus 15 at.% aluminum samples are shown in Fig. 2. It is seen that all the K_g's follow a T^2 dependence at low temperatures, indicating that dislocations are generated and arranged in a random manner. The differences in K_g between the fatigued samples (except B6) and the annealed sample are very slight and cannot be displayed clearly on such a plot. Estimates of the dislocation resistivities were made with the lattice conductivity for each sample plotted on a separate graph. The results are included in Table I. It is found that too with the accuracy of the experiment samples B2-B4 have the same dislocation density. This was initially interpreted as the saturation effect reported by Lomer and Rosenberg.[14] But upon further cycling, it is found that (sample B6) K_g decreases again, suggesting that there are further dislocation generation at this later stage of fatiguing.

This behavior appears to be peculiar and no satisfactory explanation can be offered yet. An observation worth noting is that surface convolution becomes visible on this sample and so the cross-sectional area is not as uniform as the others. Consequently, the geometric factor is determined with less accuracy. However, an analysis showed that the uncertainty in the geometric factor can only account for half the change in the measured conductivity. More importantly, an error in the geometric factor should affect the electrical resistivity measurement in the same manner, and as shown in Table I, no significant decrease in electrical resistivity was observed.

The high temperature behavior of the copper plus 15 at.% aluminum are similar to that of the copper plus 1 at.% aluminum samples. By arguments similar to those presented above, it is not necessary to invoke vacancies and stacking faults for the interpretation of the results.

CONCLUSION

Previously, the only work that has been reported on the low temperature thermal conductivity and electrical resistivity studies of fatigued alloys is by Lomer and Rosenberg.[15] Their results on copper-zinc alloys showed that there was a saturation of dislocation densities after a few hundred cycles of fatiguing. They also observed an increase in electrical resistivity that was five to ten times higher than that which occurred if the sample was deformed to the same stress level. This increase can be attributed to two sources: stacking faults formed by the dissociation of dislocations into partials, and vacancies generated by the motions of dislocations. However, as discussed in the last section these two types of defects are not detected in this investigation. This discrepancy can be explained by the argument

that the pile-up of dislocations in the copper plus 1 at.% aluminum samples precludes possibilities of vacancy generation. As dislocation movements are restricted, mechanisms for vacancy production, such as jog motion, intersecting of dislocations of different orientations or annihilation of dislocation dipoles become ineffective. Stacking faults are not expected to be produced either, as already discussed earlier. For the copper plus 15 at.% aluminum samples, dislocation densities are low and solute concentration is high. This combination would tend to result in a low mobility of dislocations, as they are likely to be pinned by the solute atoms.

The saturation of dislocation densities reported by Lomer and Rosenberg are also not demonstrated conclusively in this investigation. As it is mentioned in the last section, there is a sudden increase in dislocation density between fatiguing the 15 at.% aluminum sample to 10^5 cycles and 2.10^5 cycles. Dislocation saturation in the 1 at.% aluminum samples are observed; but the rate is not as rapid as reported by Lomer and Rosenberg.

In conclusion, results of this investigation suggest that high cycle fatigue damage in copper plus 1 at.% aluminum is predominantly in the form of dislocation pile-ups. The observation that the temperature variation of K_g at low temperatures is the same for samples at various stages of fatiguing suggests further that the cumulative damage follows a process of continued dislocation production and pile-up over a progressively larger portion of the sample. These pile-up groups of dislocations can produce severe stress concentrations at their heads and lead to the initiation of microscopic cracks, eventually causing failure. In this respect, fatigue damage does not seem to be of a different nature than that of tensile deformation, the only salient feature being that the pile-up appears to be more intense in

tensile deformation. This feature is not entirely unexpected as some of the dislocations in the pile-up may have relaxed back as stresses are reversed during cycling. These conclusions are consistent with the observations of Wood et.al.[16] who observed that for low amplitude, high cycle ($>10^6$) fatigue, isolated pores begin to appear inside the metal at 1/1000 of the fatigue life. These pores multiply on continued fatiguing, and eventually can link together to form cavities.

For copper plus 15 at.% aluminum, fatigue damage appears to be in the form of a random distribution of dislocations. One may still argue that due to the low dislocation density, the effect of possible pile-up in this alloy does not manifest itself in these measurements. Indeed, Mitchel et. al.[17] have observed pile-ups in copper-aluminum alloys with aluminum content up to 8.5 at.% . Furthermore, the electron microscopy studies of Feltner and Laird[18] have shown that the dislocation structure in specimens of copper plus 7.5 wt.% (16 at.%) aluminum that have been cyclicly strained after being annealed earlier is in the form of planar arrays or discrete bands, with the interband spacing a decreasing function of increasing strain. These bands are formed in one slip plane and dislocations in the bands tend to remain on the same slip system. However, their specimens have been subjected to a higher strain (fatigue life 10^4 cycles) and therefore the dislocation structures are more prominant. In a later paper[19] they concluded that dislocation debris is predominant in low amplitude fatigue of the same alloy. Therefore, reason for fatigue crack formation is still not clear.

Finally, in an attempt to relate the dislocation structure in a material to its slip character and the strain amplitude, Feltner and

Laird[19] seemed to suggest that for the copper plus 1 at.% aluminum alloy fatigued at low amplitudes, dislocations should be in the form of loops and dipoles. This conclusion has not been borne out by results of this investigation.

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Table I: Resistive Properties of Copper-Aluminum Samples

Sample	Cold work history	ρ μohm-cm				$W_g T^2$	$W_d T^2$	N
		1.1° K	4.2° K	77° K	273° K	(cm-deg^3/watt)		(cm^{-2})
C1	Annealed 1273° K	1.066	1.066	1.302	2.670	$1.1\ 10^{+3}$		
C2	Fatigued* 500 cycles	1.071	1.067	1.301	2.664	$1.8\ 10^{+3}$	$0.7\ 10^{+3}$	$7.8\ 10^{10}$
C3	Fatigued* 10^4 cycles	1.069	1.069	1.304	2.663	$2.0\ 10^{+3}$	$0.9\ 10^{+3}$	$1.0\ 10^{11}$
C5	Deformed 5%	1.066	1.066	1.294	2.660	$2.2\ 10^{+3}$	$1.1\ 10^{+3}$	$1.2\ 10^{11}$
C6	Fatigued* 10^5 cycles		1.064	1.306	2.665	$2.1\ 10^{+3}$	$1.0\ 10^{+3}$	$1.1\ 10^{11}$
B1	Annealed 1273° K	7.868	7.867	8.253	10.19	$2.7\ 10^{+3}$		
B2	Fatigued# 500 cycles	7.850	7.853	8.250	10.16	$2.9\ 10^{+3}$	$0.2\ 10^{+3}$	$7.4\ 10^{8}$
B3	Fatigued# 10^4 cycles	7.806	7.806	8.204	10.10	$2.9\ 10^{+3}$	$0.2\ 10^{+3}$	$7.4\ 10^{8}$
B4	Fatigued# 10^5 cycles	7.813	7.813	8.217	10.14	$2.9\ 10^{+3}$	$0.2\ 10^{+3}$	$7.4\ 10^{8}$
B5	Deformed 5%	7.889	7.889	8.288	10.16	$3.7\ 10^{+3}$	$1.0\ 10^{+3}$	$3.7\ 10^{9}$
B6	Fatigued# 2.10^5 cycles		7.891	8.273	10.21	$3.2\ 10^{+3}$	$0.3\ 10^{3}$	$1.1\ 10^{9}$

Note: C Cu-1at.%Al B Cu-15at.%Al
* Maximum load 6.4 Kg-mm^{-2}, estimated life 10^6 cycles
\# Maximum load 8.3 Kg-mm^{-2}, estimated life 10^7 cycles

FIGURE CAPTIONS

Fig. 1. Lattice Thermal Conductivity, K_g, of Copper plus 1 at.% Aluminum Samples, Plotted versus Temperature.

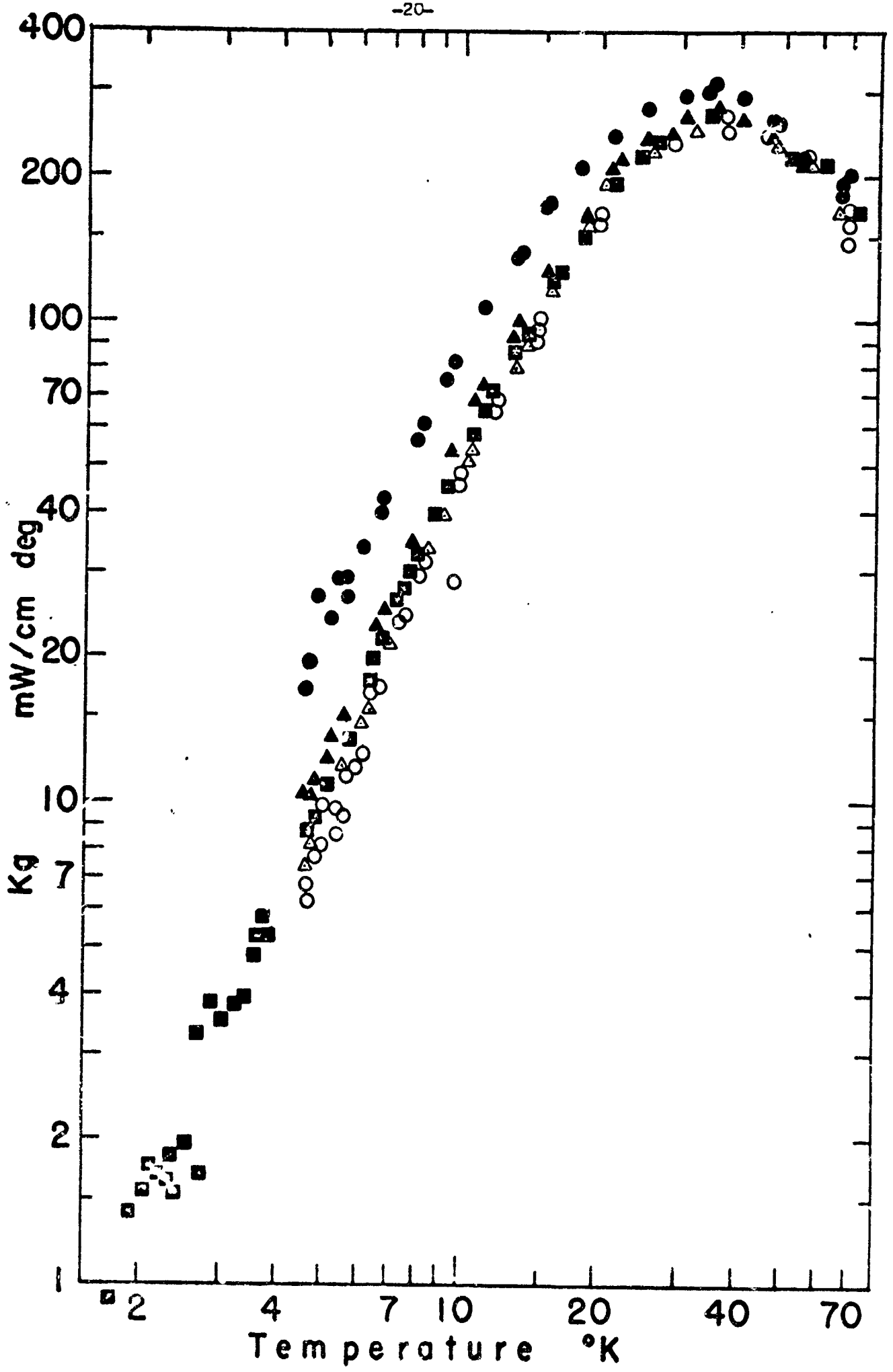
●, Sample C1. ▲, Sample C2. ■, Sample C3.

○, Sample C5. △, Sample C6.

Fig. 2. Lattice Thermal Conductivity, K_g, of Copper plus 15 at.% Aluminum Samples, Plotted versus Temperature.

△, Sample B1. ○, Sample B2. □, Sample B3.

■, Sample B4. ●, Sample B5. ▲, Sample B6.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200
100
70
40
20
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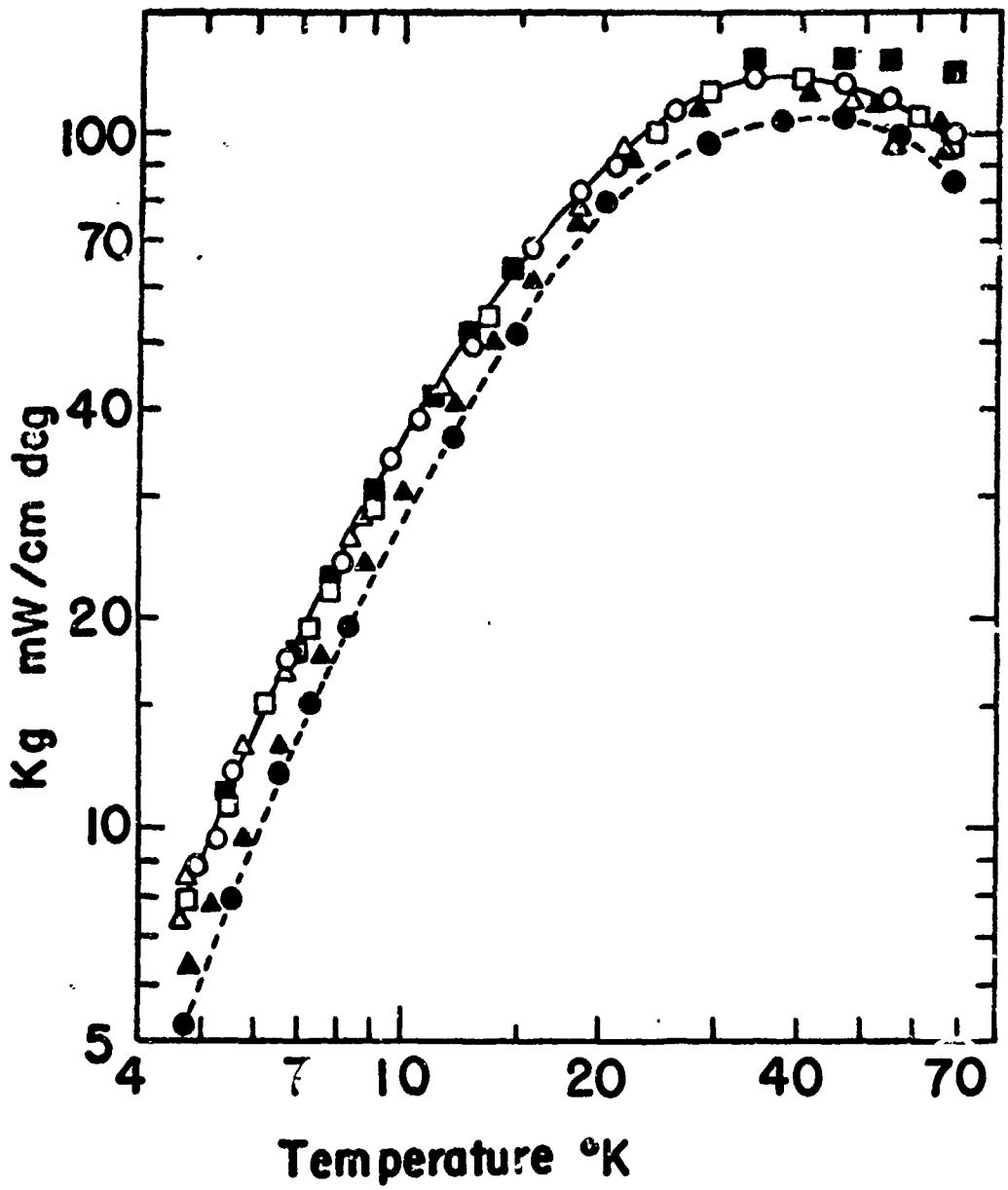
7
4
2
1
Kg mW/cm deg
2
4
7
10
20
40
70
Temperature °K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Kg mW/cm deg
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Temperature °K

INSTITUTE OF MATERIALS SCIENCE

The Institute of Materials Science was established at The University of Connecticut in 1966 in order to promote the various fields of materials science. To this end, the State of Connecticut appropriated $5,000,000 to set up new laboratory facilities, including approximately $2,150,000 for scientific equipment. In addition, an annual budget of several hundred thousand dollars is provided by the State Legislature to support faculty and graduate student salaries, supplies and commodities, and supporting facilities such as various shops, technicians, secretaries, etc.

IMS fosters interdisciplinary graduate programs on the Storrs campus and at present is supporting five such programs in Alloy Physics, Biomaterials, Crystal Science, Metallurgy, and Polymer Science. These programs are directed toward training graduate students while advancing the frontiers of our knowledge in technically important areas.